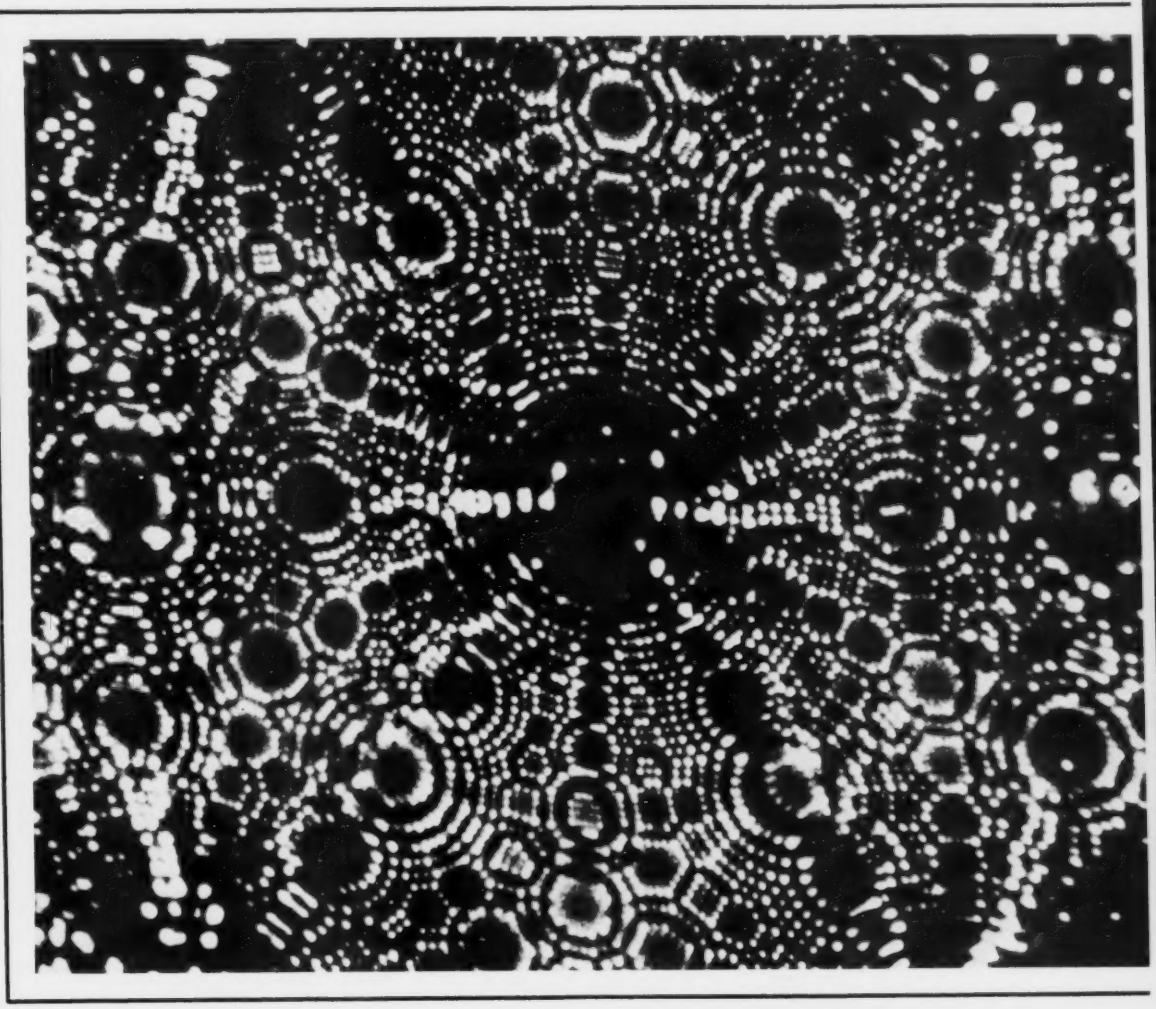
NATIONAL BUREAU OF STANDARDS October / 1965

Technical News Bulletin



U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

Technical News Bulletin

U.S. DEPARTMENT OF COMMERCE
John T. Connor, Secretary
NATIONAL BUREAU OF STANDARDS
A. V. Astin, Director

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COVER: Field-ion emission pattern of tungsten is shown with atoms appearing as bright spots. This micrograph was taken in the Bureau's field emission laboratory. A current study has shown that tungsten condensed on its own substrate will settle in the first lattice site it encounters rather than migrate to adjacent sites. (See story on page 164.)

Heat of Vaporization of HIGH TEMPERATURE METALS

TO SCIENTISTS faced with problems of missile reentry and high-speed travel in the earth's outer atmosphere, accurate data on the heat of vaporization of refractory metals are of special interest. Such data provide information on the high-temperature stability of a metal and thus play an important role in the design of space vehicles and launching equipment.

Although a number of methods are used to measure heat of vaporization, their results have not always been in good agreement, and there has been a need for some independent check on the values obtained for this quality. A method [1] that appears to meet this need has now been developed by Milton D. Scheer and Joseph Fine of the NBS Institute for Basic Standards. In this method the heated metal surface is bombarded with alkali halide molecules, and data obtained on the surface ionization of the halide are used to compute the work function of the metal. The heat of sublimation is then obtained from the temperature dependence of the self-surface ionization of the metal. As the new procedure is based on entirely different assumptions from those common to the classical methods, it should provide an excellent basis for comparing and evaluating data obtained in other ways.

Thus far, the method has been used to make heat of vaporization measurements on niobium and further work on other refractory materials is planned. The data thus accumulated will supplement those now in the Chemical Thermodynamics Data Center [2] at NBS, one of a number of centers providing input to the National Standard Reference Data System.[3]

It is well known that when a metal is heated in a vacuum, evaporation of both atoms and ions occurs. Thermodynamics shows that the heat of vaporization of a metal is a linear function of the surface work function, the first ionization potential of the metal atom, and the enthalpy of evaporation of the metal ion. Values for the ionization potential are available, and the enthalpy of ion evaporation can be obtained from an experimental determination of the current density of positive ions as a function of temperature. There remains only to determine the surface work function.

The technique used to make this determination is a modified version of a method for obtaining electron affinities. It is based on the fact that surface ionization of an alkali halide molecule (MX) yields both positive alkali (M^+) and negative halogen ions (X^-).

At a constant flux of these molecules to a heated metal surface, the ratio of positive to negative ions desorbed is dependent upon the surface temperature, work function, ionization potential of the alkali atom, and electron affinity of the halogen atom. Recent observations of the optical absorption spectra of the halogen negative ions have provided reliable electron affinities for these species,[4] and accurate alkali atom ionization potentials have been known for many years.

The procedure described above may be readily applied to a great many refractory metals. Its application to single crystal faces would be of particular interest. Since work function is known to vary with crystal orientation, it is to be expected that the enthalpy of ion evaporation, and possibly the heat of atom vaporization as well, will have a somewhat different value for each crystal plane.

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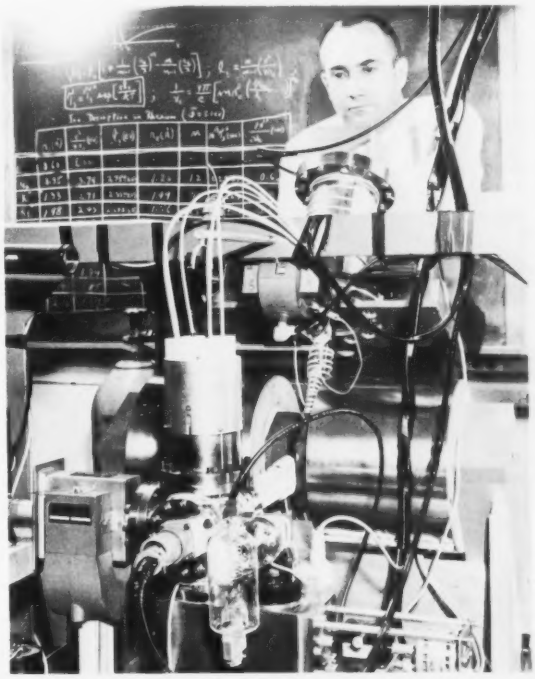
[1] *For further technical details, see* The surface ionization of niobium, *by Milton D. Scheer and Joseph Fine, J. Chem. Phys. (in press).*

[2] Thermodynamic data program, *NBS Tech. News Bull.* **42**, *No. 6, 42–108 (1958).*

[3] National Standard Reference Data System—Plan of Operation, *by Edward L. Brady and Merrill B. Wallenstein, NSRDS–NBC–1 (1964) (available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402, 15 cents).*

[4] R. S. Berry and C. W. Rieman, *J. Chem. Phys.* **38**, *1540 (1963).*



Joseph Fine adjusts the size of the exit slit in the mass spectrometer.

Ethylene-Nitrogen Atom Reaction Studied

Assumed mechanism found in error

NITROGEN ATOM reactions are of special interest to chemists studying the upper atmosphere. In studying such reactions in the laboratory it is necessary to have a means of measuring the atom concentration. The reaction of ethylene with nitrogen atoms has been extensively used as a means of measuring this concentration. However, the reaction mechanism upon which the measurement is based has now been shown to be erroneous in work done by John T. Herron of the NBS Institute for Basic Standards.[1]

The ethylene-nitrogen atom reaction is popular because of its rapidity and apparent simplicity. About 95 percent of the nitrogen in the condensable products is in the form of hydrogen cyanide, and the amount of hydrogen cyanide recovered under conditions of complete consumption of nitrogen atoms has been taken as equivalent to the quantity of nitrogen atoms originally present. That is to say, for every nitrogen atom that disappears, a molecule of hydrogen cyanide is assumed to be produced.

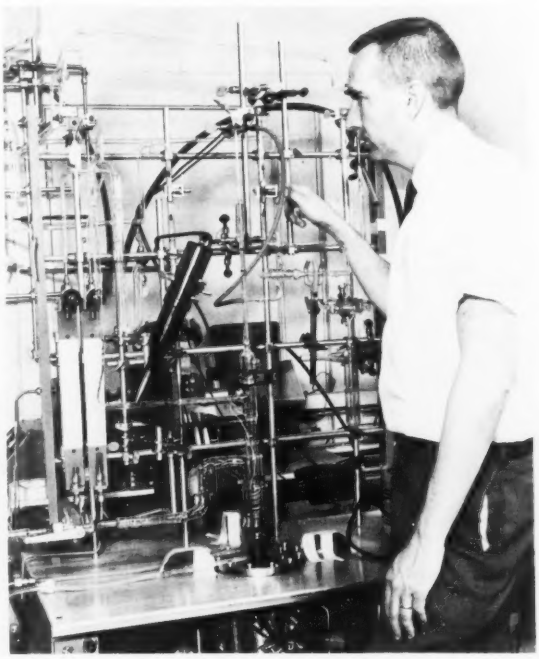
However, as was pointed out in an earlier NBS study,[2] the ratio of nitrogen atoms consumed to ethylene consumed is much too large to be accounted for by this simple mechanism. In view of these earlier results, Dr. Herron investigated the reaction in considerably greater detail in order to define unambiguously its stoichiometry, and hence to better understand its mechanism.

Because of the strong tendency of active nitrogen to recombine into the molecular (N_2) form, the partial pressure, and therefore the concentration of the atomic form of the gas, are extremely difficult to determine accurately. It was found that by utilizing new techniques of mass spectrometric partial pressure measurement, the relative partial pressure of atomic nitrogen can be accurately measured. This determination clearly indicates that there is no one-to-one correspondence between the nitrogen atoms initially present and the hydrogen cyanide molecules formed in the ethylene reaction.

To account for these observations, the following reaction mechanism is proposed:

$$N + C_2H_4 \rightarrow HCN + CH_3 \quad (1)$$
$$N + CH_3 \rightarrow HCN + 2H \quad (2)$$
$$H + C_2H_4 \rightarrow C_2H_5 \quad (3)$$
$$H + C_2H_5 \rightarrow 2CH_3 \quad (4)$$
$$N + C_2H_5 \rightarrow C_2H_4 + NH \quad (5)$$
$$N + NH \rightarrow N_2 + H \quad (6)$$

followed by the usual atom and radical recombination reactions. This series of reactions is not meant to be inclusive. It does not account for the formation of nitrogen-containing products other than hydrogen cyanide or molecular nitrogen, and it takes no account of radical disproportionation reactions, nor of radical addition reactions to ethylene.



J. T. Herron adjusts the flow of ethylene in the apparatus developed to study the reaction of nitrogen atoms with ethylene.

Recently there has been some controversy over the merits of ethylene versus nitric oxide titration techniques used to place the relative partial pressure of atomic nitrogen on an absolute basis. It would appear that this work has settled the question in favor of the nitric oxide titration.

[1] Mass spectrometric study of the reaction of nitrogen atoms with ethylene, *by J. T. Herron, J. Phys. Chem. (in press).*

[2] Rate of reaction of nitrogen atom with ethylene, *by J. T. Herron, J. Chem. Phys.* **33,** *1275 (1960).*

Improved dipole measurement characterizes OH radical

THE ELECTRIC dipole moment of the hydroxyl radical (OH) has been accurately measured [1] by F. X. Powell and David R. Lide, Jr., of the NBS Institute for Basic Standards. While this measurement does not give a value appreciably different from earlier ones, it reduces the uncertainty by an order of magnitude.

This work is part of a series of measurements designed to obtain accurate molecular constants of various radicals using the microwave technique. Furthermore, once the microwave spectrum of a material has been found, that material is uniquely characterized. The first such measurement at NBS was made on SO_2 and work is now beginning on the PN radical. The long-range goal is to investigate various unstable free radicals which are important intermediates in chemical reactions.

The difficulty of generating radicals in high concentration and the strong tendency for them to recombine quickly into more stable species are two problems which have made such measurements difficult. However, it was found that a simple reaction of hydrogen atoms with nitrogen dioxide (NO_2) yields OH in generous quantities. The recombination problem was solved by the use of an anodized aluminum waveguide coated with a fluorocarbon polymer. The new waveguide and coating were found to inhibit OH recombination far better than did the previously used stainless steel guide coated with boron oxide glass.

The measurement was carried out in a Stark-modulated free radical spectrometer.[2] The Stark field was sufficiently uniform to give well-resolved Stark components which remained well defined for large shifts. All measurements were made on the $J=7/2$ Λ doublet transition of the $^2\pi_{3/2}$ ground state.

The results of measurements made with the spectrometer lead to the value for the electric dipole moment:

$$\mu=1.660\pm0.010\ \text{D}$$

In estimating the limits of error on μ, the standard deviation was arbitrarily doubled. This uncertainty is an order of magnitude smaller than that of previous measurements of this quantity.

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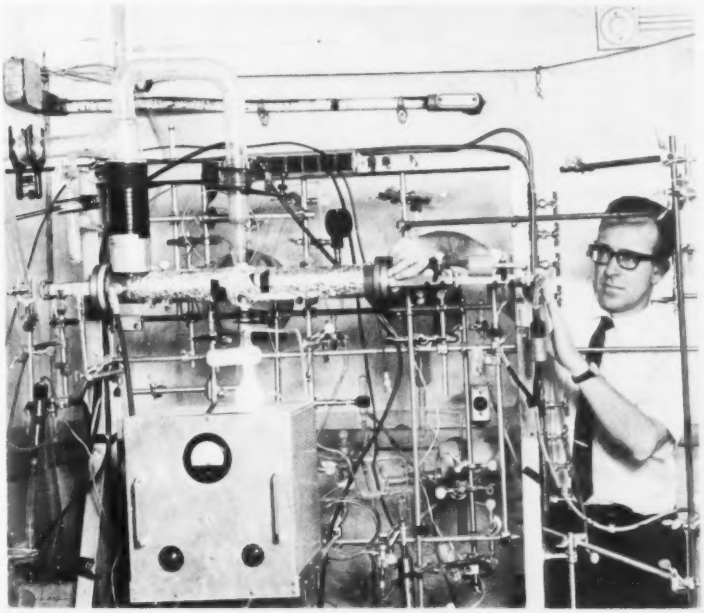
[1] *For further technical details, see* Improved measurement of the electric dipole moment of the hydroxyl radical, *by F. X. Powell and David R. Lide, Jr., J. Chem. Phys. (in press).*

[2] The microwave spectrum of the SO radical, *by F. X. Powell and David R. Lide, Jr., J. Chem. Phys.* **41**, *1413 (1964).*



F. X. Powell adjusts the waveguide structure of the free radical apparatus used to investigate the SO and OH radicals.

Effect of temperature on

OPTICAL WINDOWS

A STUDY to determine the effect of temperature on optical windows—materials that transmit certain wavelengths of light and absorb others—was recently made by A. H. Laufer, J. A. Pirog, and J. R. McNesby of the NBS Institute for Basic Standards.[1] A relationship between crystal transmittance and temperature has been known to exist but has received very little attention. Now, however, with experiments in photochemistry being conducted at high temperatures, this relationship assumes new importance.

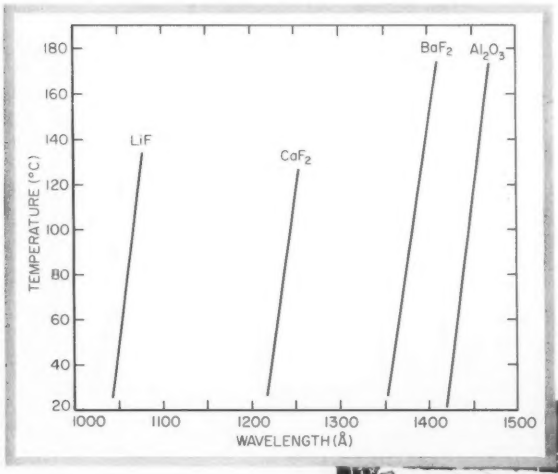
In this work, which was sponsored by the U.S. Atomic Energy Commission, the transmittance characteristics of lithium fluoride, calcium fluoride, barium fluoride, and synthetic sapphire (the alpha form of Al_2O_3) were studied as a function of temperature. It was found that, as the temperature was increased, transmittance at the short-wavelength edge of the ultraviolet spectrum was sharply reduced and the cutoff, defined as the wavelength at which transmittance is 10 percent, shifted to longer wavelengths.

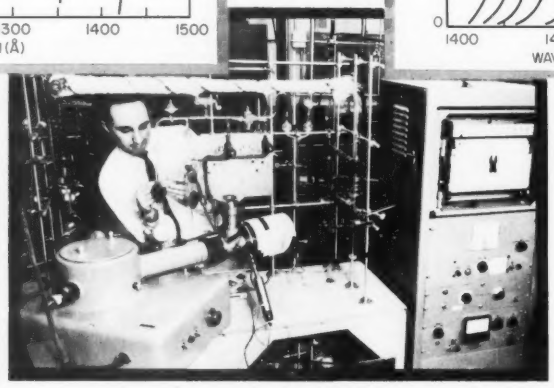
Measurements were made with a 50-cm-radius Seya-Namioka scanning monochromator with entrance and exit slit widths of 100μ. The light source was a discharge tube filled with hydrogen at 7 mm Hg pressure. The window samples were clamped in a hollow copper block (which served to maintain a constant temperature) in such a way that they could be rotated into and out of the light beam. In this manner, the incident and transmitted light intensities could easily be measured by placing a photomultiplier in the path of the light behind the sample. Window temperatures were measured by thermocouples sealed into the block.

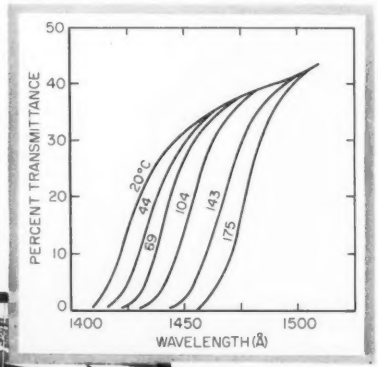
This temperature effect is of considerable importance in photochemistry. Most recent photochemical studies

(*Continued on p. 170*)

Left: *Low-wavelength transmittance cutoff as a function of temperature.* Center: *A. H. Laufer adjusts the flow of hydrogen into the light source of the apparatus used to determine the effect of temperature on the vacuum-ultraviolet transmittance characteristics of several common optical windows.* Right: *Synthetic sapphire (αAl_2O_3; 0.32 mm thick) transmittance isotherm as a function of wavelength*







Accurate Reflectance Measurements:
An ellipsoidal mirror reflectometer

AN ELLIPSOIDAL mirror reflectometer for accurate measurements on engineering materials was recently designed and constructed at the NBS Institute for Applied Technology under the sponsorship of the Air Force and the National Aeronautics and Space Administration. This instrument is the work [1] of S. T. Dunn, J. C. Richmond, and J. A. Wiebelt.*

Although the new reflectometer presently is less convenient to use than others, it is more accurate and versatile than those now in use. It may be used to measure bidirectional reflectance, directional hemispherical reflectance, the specular and diffuse components of reflectance, and directional annular cone reflectance.

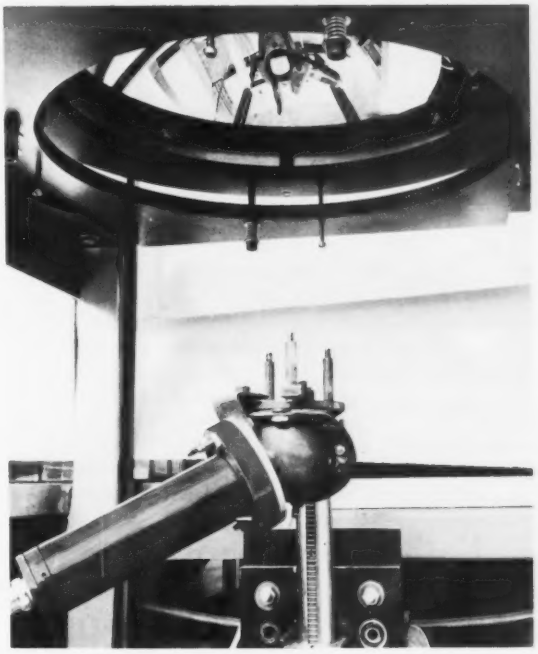
The demand for accurate and well-characterized infrared and solar reflectance data is relatively new and intimately tied to the space exploration program, where the basic problem of satellite temperature control has not been adequately solved. In addition to the space program needs, there is a continuing demand for accurate experimental verification of existing theories concerning the relations between reflectance and surface parameters such as roughness, damage, contamination, and temperature.

A lack of accurate, well-characterized data forces heat transfer engineers to assume either perfectly diffuse reflection or emission, or to assume specular reflection. Unfortunately, the most useful approach, that of assuming partial specular and partial diffuse reflectance, cannot be used, as few data of this nature are available.

Instruments presently used for reflectance measurements have several common disadvantages: restricted versatility, unknown or poorly known accuracy of measurement, and lack of absolute reflectance standards. The ellipsoidal mirror reflectometer was developed to minimize these problems.

The ellipsoidal mirror reflectometer has two major components. The first is the ellipsoidal mirror 12¼ in. in diameter and 3⅝ in. high. One focal point is in the plane of the mirror's edge and the second is 17 in. beyond the first. The other component is a detector which views the interior of a sulfur-coated averaging sphere.

Two methods may be used to determine directional hemispherical reflectance with this instrument. In the first method, absolute directional hemispherical reflectance is computed as the ratio of incident flux, measured at the first focal point, to the reflected flux, measured at the second focal point. To accomplish this, the detector is placed at the first focal point to measure the incident flux. Then a sample is placed at the first focal point and the reflected flux is focused at the second focal point by the ellipsoidal mirror where it is measured by the detector. This method requires accurate knowledge of all system losses. In the second method, relative directional hemispherical reflectance is determined by comparing the flux reflected by a sample to that reflected by a specular reflectance standard.



Two major components of the ellipsoidal mirror reflectometer are the ellipsoidal mirror (top) *and the detector which views the interior of the sulfur-coated averaging sphere.*

Other reflectances can be measured because the unique optical properties of the mirror permit accurate description of the reflected flux distribution. This is possible because the spatial distribution of reflected energy crossing the first focal plane is precisely related to the geometric distribution of the reflected flux.

(*Continued on p. 170*)

Secretary of Commerce J. T. Connor addressing the formal opening session of the National Conference.

50th National Conference on Weights and Measures

HIGHLIGHT of the 50th National Conference on Weights and Measures (June 21–25) in Washington, D.C., was the "Golden Anniversary" Exposition, a display of weights and measures exhibits, held in conjunction with the Conference. The theme of both events—Weighing and Measuring, Yesterday, Today, and Tomorrow—traced the chronology of weighing and measuring systems back to prehistoric times when cavemen probably used their own heights to measure the adequacy of their abodes.

Contrasted to the accuracies of such measurements were the accuracies in length determinations achieved today by the Bureau to 7 parts in a hundred million by laser techniques. Future weighing and measuring requirements on other planets and in the "weightless" environment of space were topics treated during the Conference.

The National Conference of Weights and Measures, organized in 1905 and held annually except in times of war or other national emergency, is sponsored by the Bureau. It provides a common meeting ground for all levels of government—Federal, State, county, municipal—concerned with weights and measures administration, and for manufacturers of weights and measures equipment and producers offering their merchandise in weighed and measured quantities.

Model laws, specifications, tolerances, regulations, and enforcement practices are recommended by the conference for adoption by the States, which are legally responsible for controlling commercial transactions involving quantity within the States. NBS, through its Office of Weights and Measures, cooperates in this endeavor by providing guidance as to reference standards and a wide range of technical, educational, and advisory programs. The reference standards are calibrated against the national standards of length, mass, and volume, which are maintained by NBS.

The 926 persons registered at the present conference comprised by far the largest number ever to attend. In addition to the official delegates from 41 States, the District of Columbia, and the Commonwealth of Puerto Rico, foreign visitors and observers from Canada, England, Germany, and Mexico, and numerous representatives from instrumentation and manufacturing industries were present.

Secretary of Commerce J. T. Connor addressed the opening session; he conveyed a message from President Johnson emphasizing the increasingly important role played by the conference in achieving uniformity in weights and measures systems throughout the country on a voluntary basis. Dr. A. V. Astin, NBS Director and Conference President, then brought the attendees up to date on recent technical developments at the Bureau in the field of measurement. Dr. D. A. Schon, director of the NBS Institute for Applied Technology, reported on measurement requirements in a dynamic society.

Dr. Allen V. Astin, President of the Conference and Director of the National Bureau of Standards, is shown cutting the ribbon to open the exposition.

Interesting statistics on the mail volume—based almost entirely on weight—handled by the U.S. Post Office Department were given by A. J. Coffman, Deputy Assistant Postmaster General. During fiscal year 1964 this volume reached the unprecedented total of almost 70 billion pieces of mail, representing one piece of mail each day for each person in the country. There are today over 45,000 individual postal facilities in the United States utilizing over a quarter of a million scales to handle this volume. Mr. Coffman detailed the progress currently being made in maintaining the accuracy of postal scales.

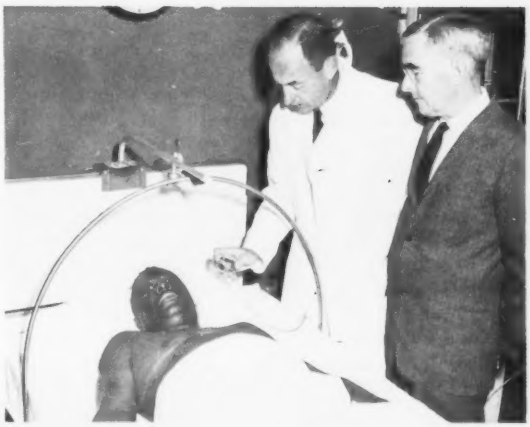
Lewis Barnard, Jr., chairman of the Board, Lufkin Rule Co., carried out the theme of the Conference in his talk on length measurements. He described the Royal Egyptian Cubit (generally recognized as the first official standard of linear measurement) designed some 5,000 years ago and based on the length of the reigning pharaoh's forearm and palm. Throughout recorded history, he said, there is ample evidence that parts of the human body were first used as units and standards.

Mr. Barnard pointed out that, in the 13th century under King Edward I of England, the strip of land plowed by a team of oxen in one day (40 rods long and 4 rods wide) was legalized as an acre. The term was probably derived from the Anglo-Saxon word "aecer," which meant a plowed or seeded field. An acre in the United States today has the same area as that decreed almost seven centuries ago. Mr. Barnard pleaded a strong case for future worldwide uniformity in units and standards of measurement, even though he agreed that serious obstacles existed to achieving such uniformity. He said, however, that there would "never be a better time than now to start trying to solve this problem."

The measurement of liquids, "yesterday" was discussed by Paul Renfrew, Liquid Controls Corp.; "today" by R. H. Tolson, Texaco, Inc.; and "tomorrow" by E. F. Wehmann, Neptune Meter Co. The science of weighing, "yesterday" was covered by W. A. Scheurer, Exact Weight Scale Co.; "today" by C. G. Gehringer, Hobart Manufacturing Co.; and "tomorrow" by V. C. Kennedy, Jr., Streeter-Amet Co.

M. D. Smith, National Canners Association, discussed the art of merchandising; and W. S. Fuller, H. J. Fuller and Sons, Inc., reviewed the philosophy of the National Scale Men's Association. W. A. Crawford, E. I. Dupont de Nemours and Co., described the Instrument Society of America and how its membership is concerned with accurate measurement systems. J. P. McBride, past chairman of the conference and retired director of standards for the State of Massachusetts, reviewed the history of the conference from the time of its inception 60 years ago. At that time, only seven States were officially represented and weights and measures requirements were far simpler than they are today.

Electrical device shows patient's pulse



NBS engineer Merlin Davis watches Dr. Edward D. Freis, of the Mt. Alto Veterans Administration Hospital (Washington, D.C.), apply an improved pulse-sensing device to patient E. L. Mitchell. The sensor was designed by Mr. Davis at NBS in a V.A.-supported program for the development of new techniques for measuring blood pressure and flow. The device's signal is produced by semiconductor strain gages bonded to a diaphragm which flexes with movements in the tissue against which the device is pressed. Here it will be positioned over an artery to study the patient's pulse, which will be automatically recorded on a chart. The sensor will be useful for diagnosis, as of aging processes and cardiovascular disorders affecting elasticity of arterial walls, and in physiological and pharmacological research.

Migration-Free Condensation Observed in Tungsten

Atoms are bound at first contact

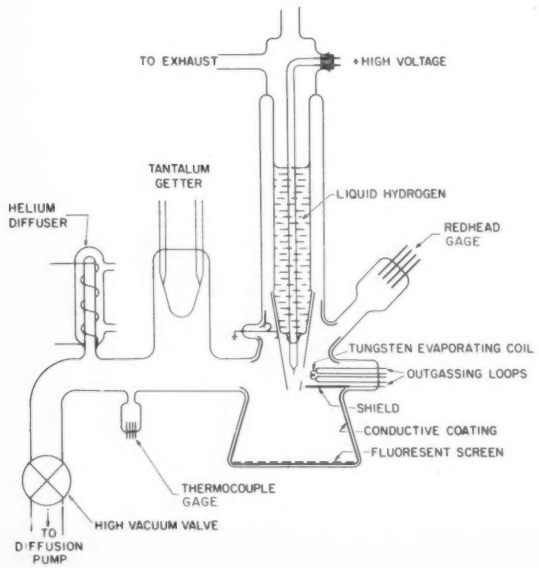
THE CONDENSATION of atoms onto single-crystal surfaces is receiving widespread attention because of its basic importance to the understanding of surface phenomena. Surface interactions that take place during the process of condensation are being studied by scientists who hope to gain a better understanding of the atomic structure of matter.

A recent study [1] has shown that, contrary to what might be expected, condensing atoms do stick in the first lattice site they encounter—at least in the case of tungsten condensing on its own substrate at temperatures below 77 °K. The study was under the sponsorship of the Advanced Research Projects Agency, the National Institutes of Health, and the American Cancer Society, and was carried out by Theodore Gurney [2] and R. D. Young at the NBS Institute for Basic Standards in cooperation with Franklin Hutchinson of Yale University.

In the case of tungsten deposited on tungsten, condensing atoms approach a surface with only a few tenths of an electron volt of energy, but, because of the strong attractive force near the surface, strike with a much greater energy—the kinetic energy of collision. In order for the atom to stick in the first lattice site it encounters, it would have to give up over half of its total energy in the first collision with the substrate. Such an occurrence would seem unlikely, so that the possibility of an atom making several lattice jumps before coming to rest could be considered quite likely. The results of the present work, however, show that migration-free condensation occurs.

An ultra-high-vacuum (better than 10^{-10} torr [3]) field ion microscope, baked to greater than 400 °C, was used in the initial study. The advantages of clean single-crystal surfaces, high resolution (better than 3 Å), and visual observation render this tool ideal for such an investigation.

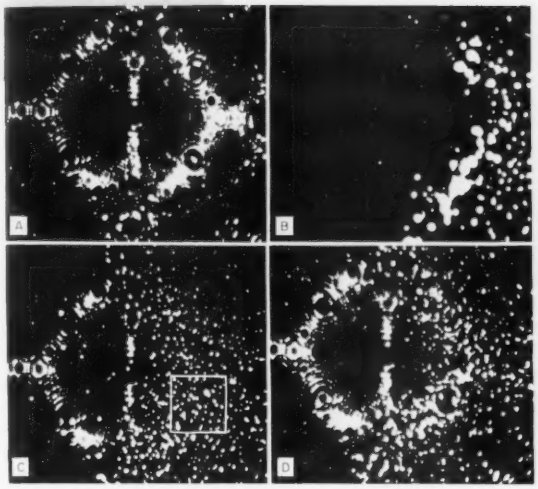
Right: *Field-ion micrographs of tungsten show various st of the condensation process.* (a) *Clean tungsten surface sh ing individual atoms in well-ordered planes.* (b) *Tung surface atoms after tungsten deposition. The large br spots are third-layer deposited atoms. Only one side of micrograph shows bright atoms because the deposited tung was "shadowed" on, i.e., was deposited from one side, thus hit only a solid-angle fraction of the emitter tip,* (c) *Surface some field evaporation. The deposited atoms seen are assu to be in the second layer. The square shows that area ch for comparison with the Monte Carlo and statistical cal tions.* (d) *First-layer deposited atoms on the surface more field evaporation.*



Tungsten was evaporated onto the tungsten field emitter by means of an evaporating coil, and the deposited atoms were then field evaporated in several steps. Photographs of the field emission pattern were taken before and after the tungsten deposition and after each field evaporation, in order to provide a step-by-step visual representation of the entire process.

Very bright spots were seen in photographs taken soon after deposition. In later photographs these bright spots were replaced by clusters of spots of lesser intensity. Apparently the brighter spots were second- or third-layer deposited atoms sitting at the apex of small clusters of atoms in the layer beneath.[4] Study of successive photographs led to the conclusion that the early pictures (taken after deposition) showed third-layer deposited atoms, later pictures (taken after some field evaporation) showed second-layer deposited atoms, still later pictures (taken after more field evaporation) showed first-layer deposited atoms, and the last pictures showed substrate atoms after all of the condensed tungsten was field evaporated.

Bakable field-ion microscope used to study the condensation of tungsten on its own substrate. Note the coil for evaporating tungsten atoms.



Under the assumption that this conclusion was correct, an atom count was made from the photographs and it was found that the third deposited layer contained only a few atoms, the second deposited layer contained only several tenths of a monolayer, and the first deposited layer was only ⅔ to ¾ filled. These findings suggested that condensing atoms were bound in the first atom site they encountered, but this explanation was considered unlikely, so further verification was sought.

Dr. Young and NBS physicist D. C. Schubert performed a Monte Carlo calculation [5] using an idealized rectangular grid of 400 atom sites (potential wells) equally spaced in the lattice. Atoms were assumed to arrive randomly at the sites. If a site was occupied, the atom was then randomly deflected to an adjacent site, unless the adjacent site was occupied, in which case it was further deflected to another nearby site.

Fractional coverage of individual atom layers as a function of total monolayer deposition. The graph shows the results of an IBM 7094 computer program, and indicates that tungsten-on-tungsten deposition at 77 °K is migration-free.

The calculation was performed for two separate cases and at least one monolayer (400 atoms) was deposited in each case. In the first case the atoms were restricted to settle in the first lattice site they encountered, while in the second case the atoms were permitted two random jumps before settling. Atoms were deposited in $\frac{1}{10}$-monolayer steps.

In order to compare the calculation results with the experiment, a fairly uniform portion of the deposited layer was selected (from the photographs) as having approximately the same area as that employed in the calculation. By comparing the number of third-layer atoms and the number of missing first-layer atoms in each calculation with the experimental results, it was easily concluded that the "no-jump" calculation was a fairly accurate approximation of the experimental results, while the "two-jump" calculation showed almost no similarity.

A more detailed understanding of the process of condensation was then obtained from a statistical calculation [6] programmed for operation on the IBM 7094 computer. Results were printed out at $\frac{1}{10}$-monolayer intervals up to a total deposition of four layers, for zero-, one-, and two-jump cases. This calculation gave an excellent picture of the details of the deposition process. In the ranges where the Monte Carlo calculation was available for comparison, numerical agreement was good except in the third layer of the no-jump case.

The outstanding feature of both calculations is the drastic lowering of population in the second and third layers when the rules are changed from zero allowed jumps to one allowed jump and then to two allowed jumps. The force of the argument lies not in the perfection of the model used for analysis, but in the drastic way in which the character of the deposit changes with the number of jumps the atom is permitted to make.

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[1] *For further technical details, see* Condensation of tungsten on tungsten in atomic detail observation with the field ion microscope, *by T. Gurney, F. Hutchinson, and R. D. Young, J. Chem. Phys.*, **42**, *No. 11 (1965).*

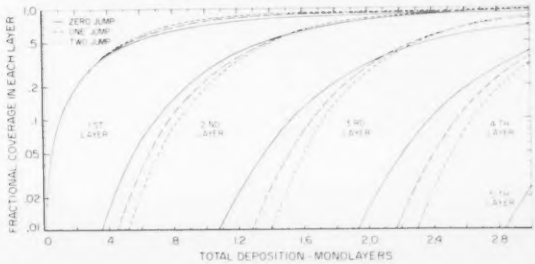
[2] *Present address, Biology Laboratory, Massachusetts Institute of Technology, Cambridge, Mass.*

[3] *1 torr=1/760 standard atmosphere=133.322 newtons/meter².*

[4] *The word "layer" refers to the height of atoms above the substrate and does not imply continuous coverage across the multifaceted surface.*

[5] *For further technical details, see* Condensation of W on W in atomic detail: Monte Carlo and statistical calculations vs. experiment, *by R. D. Young and D. C. Schubert, J. Chem. Phys.* **42**, *No. 11 (1965).*

[6] *Op. Cit., R. D. Young.*



Simplified Method for Making Quarter-Wave Plates

MANY OPTICAL INSTRUMENTS employ polarized light and associated birefractory plates as essential elements in their operation. For example, a pair of quarter-wave plates is used in photoelastic stress analysis to observe a system of isochromatic lines in a stressed plate irrespective of the azimuth of the plate. Also, quarter-wave plates are used in the Friedel method of determining optical path-difference. This method is extremely precise and has been adapted in constructing an interference miscroscope. The full advantage of the Friedel method is realized only when the optical retardation of the compensator used is exactly one quarter-wavelength.

Quarter-wave plates are usually cleaved from mica or cut from crystal quartz. These traditional methods give precise quarter-wave plates, but they are time-consuming and costly. Another method is to utilize a suitable combination of birefractory plates of arbitrary phase lag. However, until now no complete mathematical analysis has been presented for determining the conditions under which the plates could be combined to produce a "combination" quarter-wave plate.

In studying the photoelastic properties of crystals and glasses at the NBS Institute for Materials Research, a mathematical method of describing these conditions was developed. In this work, L. H. Adams and R. M. Waxler investigated the problem of making "combination" quarter-wave plates and stated the solution in a simple and convenient form.[1]

The problem is to determine what angle between the fast-ray directions of the two plates will cause the phase lags of the plates to combine to produce a phase lag of exactly one quarter-wave in the composite plate. This angle may be determined by use of the two equations below, provided the fast-ray directions and phase lags of the individual plates are known. Let the fast-ray directions and phase lags for plates I and II be α_I' and α_{II}', and Φ_I and Φ_{II}, respectively. The orientation angle, Ψ, between α_I' and α_{II}' is then found by solving the following equations:

$$\sin 2\Theta = \pm \frac{\cot \Phi_{II}}{\tan \Phi_I}, \text{ and}$$
$$\Psi = 45° - \Theta.$$

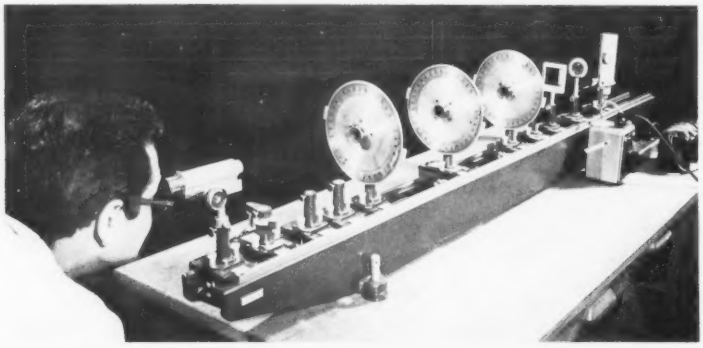
From the equations, two solutions are possible. When Ψ is positive, it is measured counterclockwise from α_{II}' to α_I'; when Ψ is negative, it is measured clockwise from α_{II}' to α_I'.* Both solutions will produce a combination quarter-wave plate. Values of Ψ have been tabulated from the equations for equal values of Φ_I and Φ_{II} at 10° intervals from 45 to 135°.

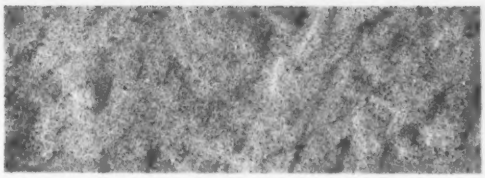
In the present study several "combination" quarter-wave plates were constructed from sheets of commercial plastic that exhibit double refraction. These combination plates have been found to perform as ordinary quarter-wave plates in the following important respects: (1) There is an angle of 45° between the azimuths of two beams of incident plane-polarized light where one beam emerges still as plane-polarized light and the other emerges as circularly polarized light. (2) The azimuth of the emerging light is independent of the azimuth of the incident plane-polarized light. (3) The ellipticity of the emerging light is equal to the angle between the azimuth of the incident plane-polarized light and one of the principal axes of the "combination" plate.

The "combination" quarter-wave plates differ from ordinary quarter-wave plates in one important respect: they exhibit optical activity superimposed upon the double refraction. This property is commonly noted in all combinations of double-refracting plates but does

(*Continued on p. 170*)

R. M. Waxler checks the performance of a combination quarter-wave plate prepared at NBS. The elements on the optical bench are (from right to left): *light source, collimating lens, optical filter, polarizer, quarter-wave plate, analyzer, and viewing telescope.*





Optical Constants of Iron Redetermined

H. T. YOLKEN and Jerome Kruger of the NBS Institute for Materials Research recently determined the optical constants of an iron single crystal surface as a function of wavelength in the visible spectrum. By a combination of experimental techniques they derived highly accurate values for iron in this spectral range. The new data will be of considerable value to theorists who are seeking additional knowledge on transitions between energy levels in metals.[1]

The optical constants of metals vary with wavelength and are not easily determined. The refractive index of the thin oxide films adhering to metal surfaces affects measurement accuracy; it distorts experimental values of the metal's optical constants. To overcome this difficulty in the present work, the oxide film was reduced by heating the specimen in pure hydrogen, followed by annealing in an ultra-high vacuum. The dual treatment produced a film-free surface that could be measured with an ellipsometric technique.[2] From these measurements, the optical constant values of iron could then be calculated.

Floating zone refined iron obtained from the Battelle Memorial Institute was used for specimen preparation. A large crystal was grown from this material by a strain anneal method and cut to a surface having a low index orientation. After a final mechanical polish of the surface with diamond paste, the specimen was lightly etched with 0.1 percent nital to remove surface contamination.

In preliminary experiments, the specimen was placed in the vacuum system which had been equipped with two optically flat quartz windows. After baking, the system was brought to an ambient pressure of 5×10^{-10} torr (6.7×10^{-8} N/m^2). The ellipsometer was then positioned adjacent to the system in such a way that the angle of incidence of a monochromatic light beam, passing through the ellipsometer onto the iron surface, was 66 degrees.

To reduce the surface oxide film present on the specimen, pure hydrogen was then diffused through a heated nickel thimble into the system and the specimen was heated at 800° C for several minutes. It was then annealed in vacuum. After ellipsometer readings were taken, the entire procedure was repeated several times until constant readings indicated that the surface was clean. These readings also indicated that an equilibrium surface topography had been attained.

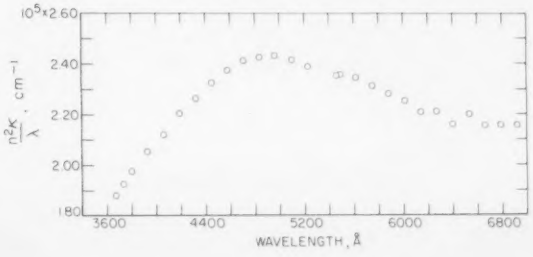
Ellipsometric measurements were then made to derive data for optical constant determinations. For these measurements, the monochromator was adjusted manually to obtain a series of readings at periodic wavelength intervals. From the resulting data, values for refractive index (n) and absorption coefficient (κ) versus wavelength were determined from 3600 to 7000 Å (360 to 700 nm).

From these values, the frequency-dependent conductivity of iron was computed as a function of wavelength. A broad maximum in the resultant curve at 4900 Å indicates interband transitions of conduction electrons in the metal, rather than the intraband transitions previously predicted by solid state theory.

[1] *For further details, see* Optical constants of iron in the visible region, *by H. T. Yolken and J. Kruger, J. Opt. Soc. Am.* **55,** *842 (1965).*

[2] *For details regarding ellipsometer operation, see* Optical measurements on thin films of condensed gases at low temperatures, *J. Kruger and W. J. Ambs, J. Opt. Soc. Am.* **49,** *1195 (1959), and* Optical properties of condensed gases, *NBS Tech. News Bull.* **44,** *171 (1960).*

Above: *This electron micrograph shows the clean surface of an iron specimen. The thin oxide film on the surface had been removed before the measurements were made by an ellipsometric technique.* Left: *H. T. Yolken adjusts a monochromator to focus green light (5490 Å) through the ellipsometer* (center and right foreground) *onto the specimen. The specimen is held in the glass vacuum cell* (right center). *A mass spectrometer in the rear is used to monitor residual gases in the system.* Below: *Values for the refractive index* (n) *and absorption coefficient* (κ) *of iron have been used to compute this graph of iron's frequency-dependent conductivity* ($n^2\kappa/\lambda$) *versus wavelength. The broad maximum that occurs at 4900 Å indicates an interband transition of conduction electrons at this wavelength.*



Simplified Method—Continued

not hinder their use. In fact, the inherent rotation provided by a "combination" quarter-wave plate may be used to advantage for the construction of certain types of polarimetric half-shades.

Precision measurements in polarimetry usually depend upon making a brightness match between two parts of a field of view. Half-shade devices permit the observer to ascertain very precisely the azimuth of plane-polarized light, and the azimuth and ellipticity of elliptically polarized light. As examples, the Lippich half-nicol and the Cornu-Jellet split nicol are widely known azimuth half-shades, and the Brace half-shade has been used extensively in the measurement of ellipticity. The results of the present study have been used to prepare four new half-shades which operate effectively as the following well-known devices: (1) A Cornu-Jellet split nicol, (2) a Laurent half-wave plate, (3) a Soliel or Nakamura biplate, and (4) a Bravais biplate. In addtion, two new half-shade devices have been prepared which are uniquely adapted to the Friedel method for the determination of optical path difference.

Most of the half-shades prepared at NBS function because of the optical activity inherent in superimposed doubly refracting plates. All of them were constructed from sheets of doubly refracting plastic, and all function adequately.

**Certain simplifications in sign conventions have been made in this article.*

[1] *For further information, see* Superimposed birefractory plates, *by L. H. Adams and R. M. Waxler, J. Res. NBS* **69C**, *No. 2, pp. 103–114 (April–June 1965).*

Optical Windows—Continued

in the vacuum ultraviolet have used lamps equipped with windows of LiF which permit polychromatic radiation to impinge upon the reaction system. For example, a krypton resonance lamp emits strongly at 1165 Å as well as at 1236 Å, and LiF also transmits both wavelengths very well. A calcium fluoride window would allow photolysis with the 1236 Å resonance line of Kr while absorbing the 1165 Å line. Elevated temperatures would render the CaF_2 window totally opaque at both wavelengths.

Thus, an obvious way to do high-temperature photochemistry at the 1236 Å line is to design a krypton resonance lamp with a water-cooled CaF_2 window and to interpose a vacuum chamber between the lamp and a reaction cell which is fitted with a LiF window. The reaction cell can be heated while the water-cooled CaF_2 lamp window transmits only the 1236 Å line. A lithium-fluoride window will transmit this radiation over a broad temperature range.

As an example of the care that must be exercised in choosing a window, the graph (p. 162, right) shows that, while sapphire is a good window for room-temperature photochemistry at 1470 Å (the xenon resonance line), it is totally unacceptable at temperatures much higher than 150 °C.

If we choose arbitrarily the minimum useful transmittance of a window as 10 percent, the relationship shown in the graph (p. 162, left) emerges. For a particular wavelength the temperature above which the window ceases to transmit more than 10 percent, may be read directly from the curve.

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[1] *For further technical details see,* Effect of temperature on the vacuum-ultraviolet transmittance of lithium fluoride, calcium fluoride, barium fluoride, and sapphire, *by A. H. Laufer, J. A. Pirog, and J. R. McNesby, J. Opt. Soc. Am.* **55**, *64 (1965).*

Accurate Reflectance Measurements—Continued

Thus, other reflectances can be measured if selectively shaped shields are placed in the first focal plane to blank out unwanted energy.

Several factors contributed to the accuracy of the instrument. The sulfur-coated averaging sphere minimized spatial and angular sensitivity. A correction technique for the entrance hole loss was effectively utilized. The ellipsoidal mirror reduced both the system aberrations and the size of the solid angle of flux incident on the detector. The effective reflectance of the mirror was measured as a function of position on the mirror; this provided an accurate correction for variations of the mirror's reflectance with position. The reflectometer needs only a specular reference standard, which is easily calibrated. System losses can be evaluated by establishing the flux involved in each loss through use of shields placed in the first focal plane.

The accuracy of this type of instrument is estimated to be at least 2 percent and probably better than 1 percent over the range of 0.4 to 10μ. A positive statement of accuracy, however, cannot be made because of the lack of comprehensive data on the goniometric distribution of reflected flux from common engineering materials. The usefulness of an accuracy of better than 1 percent has been questioned, as the condition of the surface while in use may not be definitely known. Nevertheless, this reflectometer provides more information concerning the reflectance of materials than previous instruments.

**Professor of Mechanical Engineering, Oklahoma State University, Stillwater, Okla.*

[1] *For further information, see* Design and Analysis of an Ellipsoidal Mirror Reflectometer, *by S. T. Dunn, Ph. D. Thesis, Oklahoma State University (May 1965), available from University Microfilms, Inc., Ann Arbor, Mich.*

Allen and Ambler Appointed to New Posts

Dr. Harry C. Allen, Jr., formerly Chief of the Inorganic Materials Division at the Bureau, has been named Deputy Director of the NBS Institute for Materials Research.

Dr. Ernest Ambler, formerly Chief of the Cryogenic Physics Section at NBS Institute for Basic Standards, is succeeding Dr. Allen as Chief of the Inorganic Materials Division, in the Institute for Materials Research.

The announcements were made recently by Dr. Gordon K. Teal, Director of the Institute for Materials Research. Dr. Teal said his choice was based on the fact that both men are not only outstanding scientists but possess to an unusual degree abilities that fit them for administration and management.

Dr. Allen received the Department of Commerce Gold Medal in 1964 "in recognition of highly distinguished accomplishments in research in molecular spectroscopy, and of effective leadership in the organization and administration of research programs in analytical and inorganic chemistry."

Dr. Allen has been a Bureau staff member since 1954. He is a member of the Philosophical Society of Washington, the Washington Academy of Sciences, the American Chemical Society, the American Physical Society, Phi Lambda Upsilon, and Sigma Xi. He was Associate Editor of the *Journal of Chemical Physics* from 1958 to 1960. He and Paul C. Cross, President of the Mellon Institute in Pittsburgh, Pennsylvania, and a member of the NBS Statutory Visiting Committee, are co-authors of a book entitled "Molecular Vib-Rotors, The Theory and Interpretation of High Resolution Infrared Spectra." The book presents the theory of vibrating-rotating molecules, and applies this theory to the analysis of spectral bands.

Born in Saugus, Massachusetts, Dr. Allen received his B.S. in chemistry from Northeastern University in 1948, and his M.S. in physical chemistry from Brown University a year later. He continued his studies in that field at the University of Washington where he received his Ph. D. in 1951. Dr. Allen then attended Harvard University as an Atomic Energy Commission Postdoctoral Fellow.

Dr. Ambler shared in the 1964 Samuel Wesley Stratton Award for work in demonstrating that the quantum mechanical law of parity conservation does not hold in weak interactions. He was also honored for this work with a Department of Commerce Gold Medal Award in 1957, the Washington Academy of Sciences Award in 1957, and the John Price Wetherill Medal of the Franklin Institute in 1962. In 1960, Dr. Ambler was named one of the ten outstanding young men in the Federal Government, receiving the Arthur S. Flemming Award in recognition of his outstanding work in science. In 1963 he was given the John Simon Guggenheim Memorial Foundation Fellowship Award.

Dr. Ambler, a Briton by birth, took his B.A., M.A., and Ph. D. degrees at Oxford University in 1945, 1949, and 1953. In 1953, he came to this country, joining NBS the same year. Five years later he became an American citizen.

He began performing magnetic research and nuclear orientation studies at temperatures approaching absolute zero, and within eight months after coming to the Bureau, Dr. Ambler had completed the first nuclear alinement experiments to be conducted in the United States.

Dr. Ambler is a Fellow of the American Physical Society and a member of the Washington Academy of Sciences.

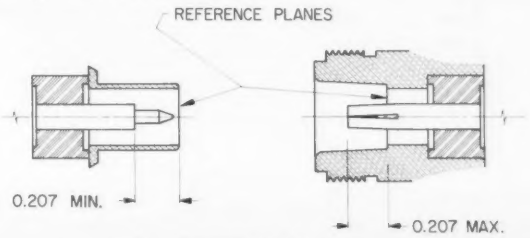
Standards and Calibration:

Attenuation calibration services for coaxial components extended to 18 GHz

THE RADIO STANDARDS Laboratory announces that calibration services for the measurement of attenuation of coaxial attenuators and couplers have been extended continuously in frequency to 18 GHz from the former range of 200 MHz to 12 GHz. Measurements are made over an attenuation range of 60 dB, with an uncertainty in measurement not exceeding 0.2 dB/10 dB for attenuators and couplers having a VSWR of 1.3 or less.

Attenuators and couplers submitted for calibration should be equipped with Type N connectors complying with the MIL C 39012 specification, or with the new precision 7 mm connectors. The critical mating dimensions used by NBS are as shown in the accompanying diagram.

Equipment fitted with connectors designed for 0.141 in. (3.5 mm) coaxial lines will be measured with an uncertainty determined by the stability and VSWR of each unit.



NOTE: DIMENSIONS IN INCHES

PUBLICATION *Briefs*

NOTE: Publications mentioned in this column, unless otherwise stated, are available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402, and through local U.S. Department of Commerce field offices. Misc. Publ. 268 $0.10 each; $6.25 per 100.

Final Volumes of Humidity and Moisture Series

VOLUMES three and four of the four-volume series, *Humidity and Moisture Measurement and Control in Science and Industry*, are now available. The series, produced under editor-in-chief Arnold Wexler of the NBS Institute for Basic Standards, deals in general with the measurement and effects of humidity. It is based on papers delivered at the 1963 International Symposium on Humidity and Moisture held in Washington, D.C.

Volume Three, Fundamentals and Standards was produced under the joint editorship of Mr. Wexler and William A. Wildhack, Associate Director of the Institute. It consists of two sections, the first, *Fundamentals*, treating definitions, nomenclature, units, transport properties, constants, and departures from ideality. The second section, *Standards*, includes methods of testing and calibrating hygrometers, humidity generators, test chambers, and saturated salt solutions. It contains data essential for testing, evaluating, and calibrating humidity measuring instruments.

Volume Four, edited by Prof. Paul N. Winn, Jr., of the University of Maryland, is devoted to *Principles and Methods of Measuring Moisture in Liquids and Solids*. It also is divided into two parts, one including descriptions of instruments and measurement methods with emphasis on such diverse methods as those using physical, chemical, dielectric resistance, capacitance, nuclear magnetic resonance, and neutron scattering techniques. Moisture measurement is described in such materials as grain, coal, rocket propellants, and soils. The other part is devoted to the interaction of moisture and materials. Topics of special interest to industry include dimensional changes due to moisture in plastic films and variation of moisture in wood.

These volumes complete the contribution to the literature of moisture and hygrometry of the 1963 Symposium, which was sponsored by NBS, the Weather Bureau, the American Society of Heating, Refrigeration, and Air-Conditioning Engineers, the American Meteorological Society, and the Instrument Society of America. Both volumes are available from the Reinhold Publishing Corp., New York, *Volume Three* for $25 and *Volume Four* for $20. The first two volumes, which appeared earlier this year, also are available from the same publisher, *Volume One, Principles and Methods of Measuring Humidity in Gases* for $30 and *Volume Two, Applications* for $27.50.

Electrical Engineering Units and Constants

Listed on convenient pocket card

THIS HANDY wallet-size card lists major electrical quantities and units, and their symbols. It also provides rounded values for physical constants most often used in the electrical field.

The list, as adopted by NBS and published in the May 1965 *Technical News Bulletin*, will provide a standard editorial usage guide for Bureau authors and publications. It is being distributed as a convenience to scientists, engineers, students, and others in electricity and electronics.

The card (Misc. Publ. 268) serves as a companion piece to NBS Miscellaneous Publication 253 ($0.05 each) which includes a more exact and comprehensive list of physical constants and a recommended list of unit prefixes.

Publication on Standardization Activities Revised

THE BUREAU is currently revising its Miscellaneous Publication 230, *Standardization Activities in the United States*, which lists over 350 American organizations engaged in standardization as a major or important part of their work.

To make the directory as comprehensive and up-to-date as possible, the Bureau requests information from all organizations in the United States which contribute to the preparation and promulgation of standards. Any organization wishing to be considered for inclusion in the publication, should send a description (200 words or less) indicating area(s) of standardization and accomplishments in the field to:

Miss Joan Hartman
National Bureau of Standards
Division 405.00
Washington, D.C. 20234

NBS Consultant Edits Radio Standards and Measurements

RADIO STANDARDS AND MEASUREMENTS, Volume I of *Progress in Radio Science 1960–1963*, is now available from its publisher. Edited by Robert W. Beatty, a radio standards engineering consultant of the Bureau, the volume contains the Proceedings of Commission I on Radio Measurements and Standards of the XIVth General Assembly of URSI at Tokyo in September 1963.

URSI (International Scientific Radio Union) is an international organization of radio scientists. Accomplishments and problems of radio science are discussed at meetings of the URSI General Assembly. URSI is organized into commissions, each assigned to study specific fields of interest to radio scientists, i.e. Commission I deals with Radio Measurements and Standards.

The new book contains papers reviewing this area of radio science from 1960 to 1963 and the current state of radio standards and measurement techniques and knowledge. The papers cover atomic and molecular frequency standards, standards broadcasts, quartz clocks, measurements and standards at frequencies to 1 GHz and at microwave frequencies, and measurements using lasers.

This progress report will be of special interest to research workers in the fields of standards and measurements and to students and educators. Copies may be obtained from the American Elsevier Publishing Co., Inc., 52 Vanderbilt Avenue, New York, N.Y., 10017 at a cost of $9.

Publications of the National Bureau of Standards

Periodicals

Technical News Bulletin, Volume **49**, No. 9, September 1965. 15 cents. Annual subscription: $1.50, 75 cents additional for foreign mailing. Available on a 1-, 2-, or 3-year subscription basis.

CRPL Ionospheric Predictions for December 1965. Three months in advance. Number 33, issued September 1965. 25 cents. Annual subscription: $2.50, 75 cents additional for foreign mailing. Available on a 1-, 2-, or 3-year subscription basis.

Journal of Research of the National Bureau of Standards

Section A. Physics and Chemistry. Issued six times a year. Annual subscription: Domestic, $4; foreign, $4.75. Single copy, 70 cents.

Section B. Mathematics and Mathematical Physics. Issued quarterly. Annual subscription: Domestic, $2.25; foreign, $2.75. Single copy, 75 cents.

Section C. Engineering and Instrumentation. Issued quarterly. Annual subscription: Domestic, $2.25; foreign, $2.75. Single copy, 75 cents.

Section D. Radio Science. Issued monthly. Annual subscription: Domestic, $9; foreign, $11.50. Single copy, $1.00.

Current Issues of the Journal of Research

J. Res. NBS **69C** *(Engr. and Instr.), No. 4 (Oct.–Dec. 1965).*

Some applications of the wave front shearing interferometer. J. B. Saunders.

Precision method for evaluating primary aberrations of lenses with a Twyman interferometer. J. B. Saunders.

Comparators for voltage transformer calibrations at NBS. W. C. Sze.

Voltage dependence of precision air capacitors. J. Q. Shields.

Single crystal x-ray diffraction at high pressures. C. Weir, S. Block, and G. Piermarini.

The Sondheimer-Wilson-Kohler formula in platinum resistance thermometry. R. J. Corruccini.

Stress analysis of tape-wound magnet coils. J. Hord.

Centerline correction for precision roughness specimens. J. L. Chamberlin.

Electric currents and potentials resulting from the flow of charged liquid hydrocarbons through short pipes. M. R. Shafer, D. W. Baker, and K. R. Benson.

A transistor screening procedure using leakage current measurements. G. T. Conrad and D. C. Shook.

Radio Sci. J. Res. NBS/URSI **69D**, *No. 10 (Oct. 1965).*

Irreversible power and radiation resistance of antennas in anisotropic ionized gases. K. S. H. Lee and C. H. Papas.

Scattering resonances of a cylindrical plasma. W. M. Leavens.

Radiation patterns from plasma enclosed cylindrical hypersonic vehicles. J. H. Harris, A. T. Villeneuve, and L. A. Broca.

The Schumann resonances. R. K. Cole, Jr.

Atmospheric radio noise bursts in the LF band at Bangalore. S. V. C. Aiya and K. N. Lakshminarayan.

Influence of finite ground conductivity on the propagation of VLF radio waves. J. R. Wait and K. P. Spies.

Model experiments on propagation of groundwaves across an abrupt boundary at perpendicular incidence. R. J. King and S. W. Maley.

Other NBS Publications

Thermodynamic and related properties of parahydrogen from the triple point to 100 °K at pressures to 340 atmospheres, H. M. Roder, L. A. Weber, and R. D. Goodwin, NBS Mono. 94 (Aug. 10, 1965), 75 cents.

Standard Reference Materials: Methods for the chemical analysis of white cast iron standards, J. I. Shultz, NBS Misc. Publ. 260-6 (July 16, 1965), 45 cents.

Guide to instrumentation literature, J. F. Smith and W. G. Brombacher, NBS Misc. Publ. 271 (July 7, 1965), $1.25. Supersedes Cir. 567.

Interrelations between cement and concrete properties, Part 1. Materials, techniques, water requirements and trace elements, R. L. Blaine, H. T. Arni, B. E. Foster, et. al., NBS Bldg. Sci. Series 2, Pt 1 (Aug 20, 1965), 35 cents

Solubility of solids in dense gases, J. M. Prausnitz, NBS Tech. Note 136 (July 1965), 35 cents.

Publications in Other Journals

This column lists all publications by the NBS staff, as soon after issuance as practical. For completeness, earlier references not previously reported may be included from time to time.

Chemistry:

Mass spectrometric study of photoionization. III. Methane and methane-d_4, V. H. Dibeler, M. Krauss, R. M. Reese, and F. N. Harllee, J. Chem. Phys. **42**, No. 11, 3791–3796 (June 1965).

Method of obtaining a range of current densities with a resistive cathode, M. Brenner and A. Brenner, Plating **56**, No. 6, 527–530 (June 1965).

National Standard Reference Data Program, E. L. Brady and S. A. Rossmassler, D. C. Libraries **35**, No. 4, 57–61 (Oct. 1964).

Negative surface ionization of complex molecules, J. T. Herron, H. M. Rosenstock, and W. R. Shields, Nature **206**, No. 4984, 611 (May 1965).

Non-equilibrium chemical excitation and chemical pumping of lasers, K. E. Shuler, T. Carrington, and J. C. Light, Apply. Opt. Suppl. **2**, pp. 81–104 (1965).

Phase equilibria studies in mixed systems of rare earth and other oxides, R. S. Roth, Book, Progress in Science and Technology of the Rare Earths, Ed. L. Ewring, **1**, 167–202 (Pergamon Press Inc., New York, N.Y. 1964).

Photolysis of ethane at the argon resonance lines 1067 and 1048 Å, A. H. Laufer and J. R. McNesby, J. Chem. Phys. **42**, No. 9, 3329–3330 (May 1, 1965).

Photoxidation of asphalts in the presence of ozone, J. R. Wright and P. G. Campbell, ACS Div. of Petroleum Chem. Preprints **9**, No. 3, 147–157 (Aug. 1964).

Precise assay of copper using small samples, T. J. Murphy and J. K. Taylor, Anal. Chem. **37**, No. 7, 929–931 (June 1965).

Pressure-induced trapping phenomenon in silver iodide, H. C. Deucker and E. R. Lippincott, Science **146**, 1295–1297 (1964).

Pyrolytic technique, G. M. Brauer, J. Polymer Sci., Pt. C, No. 8, 3–26 (1965).

Radiochemical methods of analysis (Report on Salzburg Conference), W. W. Meinke, Science, **147**, No. 3654, 182–183 (Jan. 1965).

Recommended materials and practices for use with cryogenic propellants, A. F. Schmidt, AIR **839**, 29 pages (SAE Inc., New York, N.Y., Jan. 1965).

Rotational constants of excited vibrational states of $^{14}N_2{}^{16}O$, W. J. Lafferty and D. R. Lide, J. Mol. Spectry. **14,** No. 4, 407–408 (Dec. 1964).

Second order effects in the phosphorescene of benzophenone crystals at 77 °K, R. A. Keller, J. Chem. Phys. **42,** No. 11, 4050–4051 (June 1965).

Spectrophotometric determination of the rate of dissociation of tetrafluorohydrazine behind a shock wave, L. M. Brown and B. Darwent, J. Chem. Phys. **42,** No. 6, 2158–2165 (Mar. 15, 1965).

Standardization of plastics in the United States, G. M. Kline, Courrier de la Normalisation No. 182, pp. 280–282 (Mar.–Apr. 1965); Mod. Plastics **42,** No. 9, 176 (May 1965).

Substoichiometric radiometric analysis: Determination of trace amounts of cobalt, A. R. Landgrebe, L. T. McLendon, and J. R. DeVoe, Book, Radiochemical Methods of Analysis, II, 321–333 (Intern. Atomic Energy Agency, Vienna, Austria, 1965).

The constitution of $CuFe_2O_4$, C. F. Jeerson, J. Appl. Phys. **36,** No. 3, 1165–1166 (Mar. 1965).

The gas-phase photolysis of cyclohexane in the far ultra-violet: Modes of decomposition of the neutral excited cyclohexane molecule and reactions of the parent cyclohexane ion, R. D. Doepker and P. Ausloos, J. Chem. Phys. **42,** No. 11, 3746 (June 1, 1965).

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Changes in NBS Radio Broadcasts

On September 1, 1965 there was an adjustment in the phases of time signals emitted from radio stations WWV, Greenbelt, Md., and WWVH, Maui, Hawaii. The pulses from these stations were retarded by 100 ms at 0000 UT (7:00 p.m., EST of August 31) in accordance with an announcement made by the Bureau International de l'Heure (BIH). These pulses at present occur at intervals which are longer than one second by 150 parts in 10^{10}, due to the offset maintained in carrier frequencies, as coordinated by the BIH.

On October 1, 1965 the pulses emitted from radio station WWVB, Fort Collins, Colo., were retarded by 200 ms at 0000 hours UT (7:00 p.m., EST, of September 30). The successive time pulses emitted from station WWVB are one second apart. The carrier frequency of WWVB is 60 kHz, and is broadcast without offset.

The phase adjustments ensure that the emitted pulses from all stations will remain within about 100 ms of the UT2 scale. They are made necessary because of changes in the speed of rotation of the earth with which the UT2 scale is associated. Daily UT2 information is obtained from forecasts of UT2 provided weekly by the U.S. Naval Observatory in accordance with the close cooperation maintained between the two agencies.

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